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**SULFUR CHEMILUMINESCENCE DETECTION COMPARED
TO SULFUR FLAME PHOTOMETRIC DETECTION**

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PREFACE

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SULFUR CHEMILUMINESCENCE DETECTION COMPARED TO SULFUR FLAME PHOTOMETRIC DETECTION

1. INTRODUCTION

Sulfur detection is a selective means of screening complex matrices for the presence of chemical warfare agents (CWA) bis(2-chloroethyl) sulfide (HD), bis[2(2-chloroethylthio)ethyl]ether (T), and associated analogs such as thiodiglycol and 1,4-dithiane. In the continuing effort to improve detection capability, the use of chemiluminescence has been investigated. Selected compounds were analyzed with triplicate injections and detection compared between the Sievers model 350B, Sievers model 355, and Varian flame photometric detectors (FPD). Chromatographic conditions were equivalent where possible.

Our goal for this detection capability is to provide increased sensitivity for the analysis of soils, waters, brines, and decontamination solutions for the presence of CWAs, precursors, and decomposition products. Work is currently performed using the Varian FPD. Farwell and Barinaga¹ describe the principal of this type of detection. Dyson² thoroughly describes the construction and principal of chemiluminescence.

2. EXPERIMENTATION

2.1 Instrumentation.

A Varian VISTA 6000 gas chromatograph (GC) was equipped with an FPD in the sulfur mode, on-column injection port, and a J&W, DB-608, 30 m, 0.53 mm i.d., 0.83 μ m film column with helium flowing at 10 mL/min. A Leap Technologies (Chapel Hill, NC) model CTC A200S autosampler was used to perform the 1 μ L sample injections. Data and chromatograms were generated on a Varian Instruments (Valencia, CA) 402 data system.

A Hewlett-Packard (HP) (Wilmington, DE) 5880A GC equipped with a flame ionization detector (FID) was interfaced to a chemiluminescence detector, model 355, supplied by Sievers Research, Incorporated (Boulder, CO). The GC was equipped with an on-column injection port and a Restek (Bellefonte, PA) Rtx-3 m, 0.53 mm i.d., 1.0 μ m film, or a J&W, DB-5, 30 m, 0.53 mm i.d., 1.5 μ m film column with helium flowing at 10 mL/min. A Leap Technologies model CTC A200S autosampler was used to perform the 1 μ L sample injections. Data and chromatograms were generated on an HP 5880A integrator.

An HP 5890 series II GC equipped with an FID was interfaced to a chemiluminescence detector, model 350B, supplied by Sievers Research, Incorporated. The GC was equipped with a vaporization liner in the injection port and a Restek Rtx-35, 30 m, 0.53 mm i.d., 1.0 μ m film column with helium flowing at 10 mL/min. The injection port design required a

¹Farwell, S.O., and Barinaga, C.J., "Sulfur-Selective Detection with the FPD: Current Enigmas, Practical Usage, and Future Directions," J. of Chromatographic Science Volume 24 (1986).

²Dyson, M., "The Sievers SCD 350 Detector," Analytical Proceedings Volume 30 (1993).

1 mL/min split flow and 0.5 mL/min septum purge. An HP 7673 auto injector was used to perform the 1 μ L sample injections. An HP 3396A integrator was used to generate the data and chromatograms.

2.2 GC Parameters.

Oven Temperature Initial Value: 60 °C

Initial Hold Time: 3 min

Program Rate: 10°/min

Oven Temperature Final Value: 250 °C

Final Hold Time: 5 min

Injector Temperature: 250 °C

Detector Temperature on Varian: 300 °C

2.3 Chemiluminescence Parameters.

Sievers Chemiluminescence Detector model 350

Air: 450 mL/min at pressure of 70 psi

Hydrogen: 210 mL/min at pressure of 60 psi

Vacuum at detector module 12.5 torr

Air to ozone generator set for 0.6 psi

Sievers Chemiluminescence Detector model 355

Air: 120 mL/min at pressure of 34 psi

Hydrogen: 90 mL/min at pressure of 32 psi

Vacuum at detector module 11 torr

Air to ozone generator set for 0.6 psi

2.4 Materials.

The standards were prepared from Baxter, Burdick, and Jackson capillary GC/GC/mass spectroscopy grade hexane. Bis(2-chloroethyl) sulfide was obtained from the Chemical Transfer Facility, U.S. Army Edgewood Research, Development and Engineering

NOTE: The initial temperature for ethyl methyl sulfide was 40 °C.

Center (ERDEC). The simulants, 2-chloroethyl ethyl sulfide (CEES), and O,S-diethylmethylphosphonothiolate (OS DEMP),* were synthesized by chemists in Research and Technology Directorate, ERDEC. The remaining compounds were supplied as follows:

Aldrich Chemical Company, Incorporated, Milwaukee, WI:

Ethyl methyl sulfide

Propyl sulfide

Octyl sulfide

tert-Butyl sulfide

2-Chloroethyl methyl sulfide

1,4-Thioxane

1,4-Dithiane

Dibenzothiophene

2-Thiophene acetonitrile

2-Mercaptoethanol*

2-(Methylthio)ethanol*

Alfa, Johnson Matthey Catalog Company, Incorporated, Ward Hill, MA:

Diethyl sulfide

3. DISCUSSION AND RESULTS

The concentrations of the compounds were prepared in a range of 0.5 to 20 µg/mL. The range for each compound was selected to obtain an equivalent concentration range among all compounds. Four concentrations were analyzed in triplicate for each compound. The retention times and chemical structures of the compounds are listed in Table 1.

Detector responses have been tabulated for each compound in Tables 2-7. Compound name, concentration, column type, average response, and standard deviation are tabulated. Overall examination shows the FPD could not detect sulfur at the lowest concentration range of 0.54 to 1.2 µg/mL for all compounds analyzed except 1,4-dithiane and dibenzothiophene. Sievers model 355 could not detect the same concentration range for all compounds except tert-butyl sulfide and ethyl methyl sulfide. Table 8 provides some physical properties necessary for computing sulfur concentration for compounds analyzed.

*These compounds were not used due to very poor chromatography.

The Sievers chemiluminescence detector model 350B was superior to the Varian FPD and Sievers chemiluminescence detector model 355 by linearity and sensitivity. The Sievers model 350B performed well for all of the compounds analyzed.

The Varian FPD, although not as sensitive as the Sievers model 350B, did detect all of the compounds and maintained linearity.

The Sievers model 355 did not detect the 1,4-thioxane, dibenzothiophene, or octyl sulfide. This detector was not as sensitive as the FPD for 1,4-dithiane and ethyl methyl sulfide. However, it was more sensitive for tert-butyl sulfide; otherwise, it performed the same as the FPD.

To verify that observed sensitivity differences between Sievers models was neither probe nor detector module related, we interchanged the detector units on the HP 5880 and HP 5890. The resulting differences were insignificant.

This lack of response between the Sievers models 355 and 350B is believed to be due to a degree of cooling experienced with the Sievers model 355 plasma flame versus the FID open flame construction required for the Sievers model 350B. A second assumption is the stabilization compounds experience because of functional group shield and/or surrounding electron clouds. These hypotheses require further investigation.

The chemiluminescence detector technique used to screen decontamination solutions for the presence of specific sulfur-containing compounds is very useful due to the absence of hydrocarbon interferences, which exist with FPD analysis. The technique must be coupled to a switching valve to vent halogenated solvents. Our work shows halogenated solvents drastically reduce the sensitivity upon the initial injection of any extracted sulfur analyte. Our specific work was performed with methylene chloride and chloroform. We also found that methanol and ethanol reduced the sensitivity of both the Sievers models (350B and 355). We strongly recommend the use of either hexane or any similar hydrocarbon.

The second sulfur concentration range not detected occurred with the FPD analysis of 2-thiophene acetonitrile, octyl sulfide, and methyl ethyl sulfide. The Sievers model 355 did not detect the second concentration range for analytes 2-thiophene acetonitrile, 1,4-thioxane, 1,4-dithiane, dibenzothiophene, and octyl sulfide. This detector also failed to detect all concentrations for 1,4-thioxane, dibenzothiophene, and octyl sulfide. The Sievers model 350B detected all compounds and concentrations.

Data trends indicated lower concentration values result in higher value for the standard deviation. For example, the percent standard deviation for the concentration of tert-butyl sulfide dropped from 4% for 1.701 $\mu\text{g/mL}$ to 1% at 6.804 $\mu\text{g/mL}$ for the FPD, from 7 to 3% for Sievers model 350B, and from 14 to 7% over the same concentration range. The high range on the model 350B is attributed to the difference observed in on-column injection performed on the Varian 6000 and HP 5880 versus the vaporization configuration standard on the HP 5890 Series II megabore injectors.

4. CONCLUSION

Examination based on sensitivity suggests the Sievers detectors (models 355/350B) in some cases performed better than the Varian flame photometric detectors (FPD). Overall, examination indicates the FPD is the recommended detector for screening unknown sulfur compounds. Our experiments indicate that the Sievers sulfur sensitivity is compound structure related. This is exemplified by comparison of responses measured for 1,4-thioxane on the Sievers model 355 versus 350B. Unexpectedly, the model 350B detected all compounds. It is theorized that the reduced response on the Sievers model 355 is attributed to the plasma flame this system uses. Further study is required.

TABLE 1. CHEMICAL STRUCTURE AND RETENTION TIMES
OF THE COMPONENTS

	CHEMICAL NAME	CHEMICAL STRUCTURE	RT
1	2-Chloroethylethyl sulfide	$\text{Cl-CH}_2\text{CH}_2\text{-S-CH}_2\text{CH}_3$	5.0
2	O,S-Diethylmethyl-phosphonothiolate	$\begin{array}{c} \text{O} \\ \text{CH}_3\text{-P-S-CH}_2\text{CH}_3 \\ \text{OCH}_2\text{CH}_3 \end{array}$	*
3	tert-butyl sulfide	$[(\text{CH}_3)_3\text{C}]_2\text{S}$	3.5
4	2-chloroethyl methyl sulfide	$\text{ClCH}_2\text{CH}_2\text{SCH}_3$	3.4
5	2-Thiophene acetonitrile	$\text{C}_6\text{H}_5\text{NS}$	11.6
6	1,4-Thioxane	$\text{C}_4\text{H}_8\text{OS}$	4.6
7	1,4-Dithiane	$\text{C}_4\text{H}_8\text{S}_2$	8.6
8	2-Mercaptoethanol	$\text{HSCH}_2\text{CH}_2\text{OH}$	*
9	Dibenzothiophene	$\text{C}_{12}\text{H}_8\text{S}$	18.7
10	Diethyl sulfide	$(\text{CH}_3\text{CH}_2)_2\text{S}$	1.2
11	Ethyl methyl sulfide	$\text{C}_3\text{H}_8\text{S}$	1.2
12	Octyl sulfide	$\text{C}_{16}\text{H}_{34}\text{S}$	18.1
13	Propyl sulfide	$\text{C}_6\text{H}_{14}\text{S}$	3.5
14	2-(methylthio)ethanol	$\text{CH}_3\text{SCH}_2\text{CH}_2\text{OH}$	*
15	2,2'-Dichloroethyl sulfide	$\text{ClCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{Cl}$	10.0

*These compounds were dropped from the study due to very poor chromatography.

TABLE 2. COMPARISON OF SULFUR DETECTOR RESPONSES FOR FLAME
PHOTOMETRIC AND CHEMILUMINESCENCE FOR 2-THIOPHENE
ACETONITRILE AND 2-CHLOROETHYLETHYL SULFIDE

2-Thiophene Acetonitrile												
Varian FPD				Sievers/HP 5880						Sievers/HP 5890		
Molecular Weight(5)*	DB-608 0.83 μm			DB-5 1.5 μm			Rtx-35 1.0 μm			Rtx-35 1.0 μm		
($\mu\text{g/mL}$)	x	%	n	x	%	n	x	%	n	x	%	n
0.6	-	-	-	-	-	-	-	-	-	34035	2	583
1.2	-	-	-	-	-	-	-	-	-	91338	13	11985
3.0	78	6	5	-	-	-	73	36	27	189012	6	10561
6.0	170	2	4	-	-	-	132	14	18	300142	10	30580

2-Chloroethylethyl Sulfide												
Varian FPD				Sievers/HP 5880						Sievers/HP 5890		
Molecular Weight (1)*	DB-608 0.83 μm			DB-5 1.5 μm			Rtx-35 1.0 μm			Rtx-35 1.0 μm		
($\mu\text{g/mL}$)	x	%	n	x	%	n	x	%	n	x	%	n
0.5	-	-	-	-	-	-	-	-	-	41789	3	1348
1.1	93	3	3	245	45	111	114	7	8	118613	17	19692
5.4	224	3	7	585	3	15	497	4	22	586352	5	28197
10.8	528	2	10	1099	3	29	1133	10	11	1295657	9	119045

*See Table 8 for compounds' physical properties.

TABLE 3. COMPARISON OF SULFUR DETECTOR RESPONSES FOR FLAME
PHOTOMETRIC AND CHEMILUMINESCENCE FOR TERT-BUTYL
SULFIDE AND 2-CHLOROETHYL METHYL SULFIDE

tert-Butyl Sulfide												
Varian FPD				Sievers/HP 5880						Sievers/HP 5890		
Molecular Weight (3)*	DB-608 0.83 μ m			DB-5 1.5 μ m			Rtx-35 1.0 μ m			Rtx-35 1.0 μ m		
(μ g/mL)	x	%	n	x	%	n	x	%	n	x	%	n
0.68	-	-	-	375	7	25	269	7	19	113550	4	4436
1.70	150	4	6	931	6	58	742	7	51	310947	14	44342
3.40	458	3	13	2369	2	43	1461	6	81	597715	6	37964
6.80	743	1	10	3612	2	70	3281	3	87	1166566	7	77575

2-Chloroethyl Methyl Sulfide												
Varian FPD				Sievers/HP 5880						Sievers/HP 5890		
Molecular Weight (4)*	DB-608 0.83 μ m			DB-5 1.5 μ m			Rtx-35 1.0 μ m			Rtx-35 1.0 μ m		
(μ g/mL)	x	%	n	x	%	n	x	%	n	x	%	n
1.0	-	-	-	-	-	-	-	-	-	74637	4	3071
2.0	56	11	5	37	70	26	107	35	38	112487	4	3327
4.0	134	1	2	165	23	38	161	32	15	207541*	2	5075
10.0	348	8	3	428	12	52	320	10	32	331724	3	11412

*See Table 8 for compounds' physical properties.

NOTE: For the HP 5890, the concentration of the third vial was 6 μ g/mL, instead of 4 μ g/mL.

TABLE 4. COMPARISON OF SULFUR DETECTOR RESPONSES FOR FLAME
PHOTOMETRIC AND CHEMILUMINESCENCE FOR 1,4-THIOXANE AND
1,4-DITHIANE

1,4-Thioxane												
Varian FPD				Sievers/HP 5880						Sievers/HP 5890		
Molecular Weight (6)*	DB-608 0.83 μ m			DB-5 1.5 μ m			Rtx-35 1.0 μ m			Rtx-35 1.0 μ m		
(μ g/mL)	x	%	n	x	%	n	x	%	n	x	%	n
1.15	-	-	-	-	-	-	-	-	-	70873	3	1932
2.89	154	3	5	-	-	-	-	-	-	167142	2	2695
5.77	335	0.5	21	-	-	-	-	-	-	340920	0.4	1332
11.54	741	0.3	2	-	-	-	-	-	-	664329	5	35213

1,4-Dithiane												
Varian FPD				Sievers/HP 5880						Sievers/HP 5890		
Molecular Weight (7)*	DB-608 0.83 μ m			DB-5 1.5 μ m			Rtx-35 1.0 μ m			Rtx-35 1.0 μ m		
(μ g/mL)	x	%	n	x	%	n	x	%	n	x	%	n
0.67	58	17	10	-	-	-	-	-	-	64856	4	2711
1.69	164	2	4	-	-	-	-	-	-	154719	0.5	840
3.37	345	0.3	1	-	-	-	61	28	17	320976	0.5	1540
6.74	711	0.1	1	-	-	-	194	21	41	635961	2	13802

*See Table 8 for compounds' physical properties.

TABLE 5. COMPARISON OF SULFUR DETECTOR RESPONSES FOR FLAME PHOTOMETRIC AND CHEMILUMINESCENCE FOR 2,2'-DICHLOROETHYL SULFIDE AND DIBENZOTHIOPHENE

2,2'-Dichloroethyl Sulfide												
Varian FPD				Sievers/HP 5880						Sievers/HP 5890		
Molecular Weight (15)*	DB-608 0.83 μm			DB-5 1.5 μm			Rtx-35 1.0 μm			Rtx-35 1.0 μm		
($\mu\text{g/mL}$)	x	%	n	x	%	n	x	%	n	x	%	n
0.91	-	-	-	-	-	-	-	-	-	30186	3	961
2.26	189	1	2	-	-	-	114	14	17	77757	4	2751
4.53	312	1	3	46	69	32	215	11	23	155300	0.6	949
9.05	439	1	6	147	31	46	433	1	6	301061	1	3186

Dibenzothiophene												
Varian FPD				Sievers/HP 5880						Sievers/HP 5890		
Molecular Weight (9)*	DB-608 0.83 μm			DB-5 1.5 μm			Rtx-35 1.0 μm			Rtx-35 1.0 μm		
($\mu\text{g/mL}$)	x	%	n	x	%	n	x	%	n	x	%	n
2.11	77	3	2	-	-	-	-	-	-	53139	5	2719
4.22	134	3	3	-	-	-	-	-	-	108612	4	3830
8.44	274	0.4	1	-	-	-	-	-	-	230534	2	4213
16.9	602	0.5	3	-	-	-	-	-	-	459431	2	10234

*See Table 8 for compounds' physical properties.

TABLE 6. COMPARISON OF SULFUR DETECTOR RESPONSES FOR FLAME
PHOTOMETRIC AND CHEMILUMINESCENCE FOR OCTYL SULFIDE
AND PROPYL SULFIDE

Octyl Sulfide												
Varian FPD				Sievers/HP 5880						Sievers/HP 5890		
Molecular Weight (12)*	DB-608 0.83 μm			DB-5 1.5 μm			Rtx-35 1.0 μm			Rtx-35 1.0 μm		
($\mu\text{g/mL}$)	x	%	n	x	%	n	x	%	n	x	%	n
1.17	-	-	-	-	-	-	-	-	-	17097	8	1382
2.35	-	-	-	-	-	-	-	-	-	24429	2	480
4.70	53	4	2	-	-	-	-	-	-	39344	5	2075
9.40	155	2	3	-	-	-	-	-	-	71367	3	2078

Propyl Sulfide												
Varian FPD				Sievers/HP 5880						Sievers/HP 5890		
Molecular Weight (13)*	DB-608 0.83 μm			DB-5 1.5 μm			Rtx-35 1.0 μm			Rtx-35 1.0 μm		
($\mu\text{g/mL}$)	x	%	n	x	%	n	x	%	n	x	%	n
0.84	-	-	-	-	-	-	-	-	-	40104	5	1993
2.10	141	1	2	-	-	-	232	7	16	96056	3	3255
4.21	273	1	4	-	-	-	522	11	56	165330	1	2403
8.41	656	0.5	3	-	-	-	1185	2	18	344426	11	36882

*See Table 8 for compounds' physical properties.

TABLE 7. COMPARISON OF SULFUR DETECTOR RESPONSES FOR FLAME
PHOTOMETRIC AND CHEMILUMINESCENCE FOR DIETHYL SULFIDE
AND ETHYL METHYL SULFIDE

Diethyl Sulfide												
Varian FPD				Sievers/HP 5880						Sievers/HP 5890		
Molecular Weight (10)*	DB-608 0.83 μ m			DB-5 1.5 μ m			Rtx-35 1.0 μ m			Rtx-35 1.0 μ m		
(μ g/mL)	x	%	n	x	%	n	x	%	n	x	%	n
0.71	-	-	-	-	-	-	-	-	-	23958	4	937
1.77	78	1	1	-	-	-	160	8	12	56274	2	1257
3.54	154	0.6	1	-	-	-	287	6	18	114905	13	15451
7.08	384	0.3	1	-	-	-	664	4	29	203554	3	5318

Ethyl Methyl Sulfide												
Varian FPD				Sievers/HP 5880						Sievers/HP 5890		
Molecular Weight (11)*	DB-608 0.83 μ m			DB-5 1.5 μ m			Rtx-35 1.0 μ m			Rtx-35 1.0 μ m		
(μ g/mL)	x	%	n	x	%	n	x	%	n	x	%	n
0.78	-	-	-	-	-	-	35	77	27	36390	3	1090
1.55	-	-	-	-	-	-	97	32	31	77578	3	2506
3.11	157	1	2	-	-	-	200	12	23	162882	4	6010
6.21	361	0.3	1	-	-	-	436	5	22	338485	1	3525

*See Table 8 for compounds' physical properties.

Since above data were not favorable, the compound was analyzed again. See below.

(μ g/mL)	x	%	n
0.77	144	72	125
1.55	2142	19	414
3.11	24603	4	866
6.21	109520	25	2617

TABLE 8. PHYSICAL PROPERTIES OF COMPOUNDS ANALYZED IN TABLE 1

COMPOUND NO.	MOLECULAR WT	BOILING PT	SULFUR CONTENT
1	125.0	156-157	0.256
2	168.0	223	0.190
3	146.3	147-151	0.219
4	110.6	55-56 (30 MM)	0.289
5	123.0	115-120 (22 MM)	0.260
6	104.0	147 (755 MM)	0.308
7	120.2	*	0.266
8	78.1	157-158	0.410
9	184.2	*	0.174
10	90.0	91	0.356
11	76.1	66-67	0.420
12	258.5	180 (10 MM)	0.124
13	118.2	142-143	0.271
14	92.0	169-171	0.356
15	159.0	227.8	0.201

*Compound is a solid.